

UNIVERSITY OF TORONTO



Committee of Revision and Publication
of the Pharmacopœia of the
United States of America: 1890-1900.

ALKALOIDAL ESTIMATION:

A BIBLIOGRAPHICAL INDEX OF CHEMICAL RESEARCH
PREPARED FROM ORIGINAL LITERATURE
FOR THE COMMITTEE OF REVISION

BY

PAUL I. MURRILL

UNDER THE DIRECTION OF

ALBERT B. PRESCOTT.

PUBLISHED BY THE COMMITTEE OF REVISION

ANN ARBOR.
1898.

[THIS PAMPHLET IS NOT FOR SALE.]

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BIBLIOGRAPHICAL INDEX OF ALKALOIDAL ESTIMATION.

1877-1897.

The object of this work is to furnish a descriptive index of the work that has been done on the subject, rather than to abstract or summarize it.

For convenience, as large a list of references is given for each article as is practicable, the first referring to the original publication, and the others to republications or abstracts. In preparing it, the following publications have been examined up to the end of 1897:

Journal of the Chemical Society, London.

Chemisches Centralblatt.

American Journal of Pharmacy.

Proceedings of the American Pharmaceutical Association.

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Lyon's Manual of Pharmaceutical Assaying (1886).

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American Druggist.

Analyst, (London.)

Annalen der Chemie (Liebig).

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Abbreviations used are those of Bolton's "Bibliography of Chemistry," as far as possible. Others are believed to be self-explanatory.

References have been verified as far as practicable, and fully wherever any doubt has arisen in regard to possible errors.

ALKALOIDAL ESTIMATION.

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3

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Br. Pharm. 1885, p. 111. [See 1898 (4).]

FOR QUININE AND CINCHONIDINE.—Mix 200 grains of red cinchona bark, in No. 60 powder, with 60 grs. of calcium hydrate; slightly moisten the powders with half an ounce of water; mix the whole intimately in a small porcelain dish or mortar; allow the moisture to stand for an hour or two, when it will present the characters of a moist dark brown powder, in which there should be no lumps or visible white particles. Transfer this powder to a six ounce flask, add three fluid-ounces of benzolated amyl alcohol, boil them together for about half an hour, decant and drain off the liquid onto a filter, leaving the powder in the flask; add more of the benzolated amyl alcohol to the powder, boil and decant as before, and repeat the operation a third time; then turn the contents of the flask on to the filter, and wash by percolation with more of the benzolated amyl alcohol until the bark is exhausted. If, during the boiling

a funnel be placed in the mouth of the flask, and another flask of cold water be placed in the funnel, this will form a convenient condenser which will prevent the loss of more than a small quantity of the boiling liquid. Introduce the collected filtrate while still warm into a stoppered glass separator; add to it 20 minims of diluted hydrochloric acid, mixed with two fluid drachms of water; shake them well together, and when the acid liquid has separated it may be drawn off, and the process repeated with distilled water slightly acidulated with hydrochloric acid, until the whole of the alkaloids have been removed. The acid liquid thus obtained will contain the alkaloids as hydrochlorates, with excess of acid. It is to be carefully and exactly neutralized with ammonia while warm, and then concentrated to the bulk of three fluid drachms. If now about 15 grs. of tartarated soda (sodium potassium tartrate) be added to the neutral hydrochlorates, and the mixture stirred with a glass rod, insoluble tartrates of quinine and cinchonidine will separate completely in about an hour; and these collected on a filter, washed and dried, will contain eight-tenths of their weight of the alkaloids quinine and cinchonidine, which, divided by 2, represents the percentage of those alkaloids. The other alkaloids will be left in the mother-liquor.

FOR TOTAL ALKALOIDS.—To the mother-liquor from the preceding process add solution of ammonia in slight excess. Collect, wash, and dry the precipitate, which will contain the other alkaloids. The weight of this precipitate, divided by 2, and added to the percentage weight of the quinin and cinchonidine, gives the percentage of total alkaloids.

1885: (20) BRITISH PHARMACOPŒIA. Assay of extract of nuxvomica.

Br. Pharm. 1885, p. 163. [See 1898: (6).]

Dissolve 10 grains of the extract in half a fluid ounce of water, heating gently if necessary, and add a drachm of sodium carbonate previously dissolved in half a fluid ounce of water and half a fluid ounce of chloroform, agitate, warm gently, and separate the chloroform. Add to this half a fluid ounce of dilute sulphuric acid, with an equal bulk of water; again agitate, warm and separate the acid liquor from the chloroform. To this acid liquor add now an excess of ammonia, and agitate with half a fluid ounce of chloroform; when the liquors have separated, transfer the chloroform to a weighed dish, and evaporate the chloroform over a water bath. Dry residue for an hour, and weigh.

1885: (21) BRITISH PHARMACOPŒIA. Assay of opium.

Br. Pharm. 1885, p. 296. [See 1898: (7).]

Triturate together in a mortar 140 grains dried powdered opium, 60 grains freshly slaked lime, and 400 grain-measures of distilled water,

until a uniform mixture results; then add 1,000 grain-measures of distilled water, and stir occasionally during half an hour. Filter the mixture through a plaited filter about 3 in. in diameter into a wide mouthed bottle or stoppered flask (having the capacity of about 6 fluid ounces, and marked exactly at 1,040 grain-measures) until the filtrate reaches this mark. To the filtered liquid (representing 100 grains of opium) add 100 grain-measures of rectified spirit, and 500 grain-measures of ether, and shake the mixture; then add the chloride of ammonium, shake well and frequently during half an hour, and set it aside for twelve hours. Counterbalance two small filters; place one within the other in a small funnel, and decant the ethereal layer as completely as possible upon the inner filter. Add 200 grain-measures of ether to the contents of the bottle and rotate it; again decant the ethereal layer upon the filter, and afterwards wash the latter with 100 grain-measures of ether slowly added and in small portions. Now let the filter dry in the air and pour upon it the liquid in the bottle in portions, in such a way as to transfer the greater portion of the crystals to the filter. When the liquid has passed through the filter, wash the bottle and transfer the remaining crystals to the filter, with several small portions of distilled water, using not much more than 200 grain-measures in all, and distributing the portions evenly upon the filter. Allow the filter to drain, and dry it, first by pressing between sheets of bibulous paper, and afterwards at a temperature between 131° and 140° F. (55° and 60° C), and finally at 194° to 212° F. (96° to 100° C.) Weigh the crystals in the inner filter, counter-balancing by the other filter. The crystals should weigh ten grains (not less than $9\frac{1}{2}$ nor more than $10\frac{1}{2}$), corresponding to about 10 per cent. of morphine in the dry powdered opium.

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1886: (2) SCHEIBLER. Method of purification of alkaloids, using phosphotungstic acid.

Lyons' Manual, p. 33.

1886: (3) LYONS, A. B. Estimation of berberine, colorimetric.
Lyons' Manual, p. 102.

1886: (4) LYONS, A. B. Estimation of hydrastine, gravimetric.
Lyons' Manual, p. 104.

- 1886: (5) LYONS, A. B. Estimation of physostigmine (eserine) gravimetric.
Lyons' Manual, p. 122.
- 1886: (6) LYONS, A. B. Approximate estimation of strychnine, gravimetric.
Lyons' Manual, p. 113.
- 1886: (7) LYONS, A. B. Volumetric estimation of various alkaloids with Mayers' reagent.
Am. J. Pharm. 1886, 579; 1887, 1; Proc. Mich. S. Ph. A. 1886, 167; Jsb. d. Pharm. 1887, 369; Proc. Am. Ph. A. 1887, 303; Lyons' Manual, p. 23.
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- 1886: (9) LYONS, A. B. Assay of cinchona, modification of Prollius' method.
Drug. Circ. 1886, 150.
- 1886: (10) HOOPER, D. Optical method of analysis of quinine sulphate.
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- 1886: (18) WRAMPELMEIER and MEINERT. As to a true aliquot part, by volume, of the total liquid in opium assay, and as to the solvent effect of ammonium chloride in the same liquid.
Proc. Mich. State Phar. Assoc., 4, 127; Am. Drugg., 15, 203.
- 1887: (1) PLUGGE, P. C. Alkalimetric estimation of alkaloids.
Arch. Pharm. (3) 25, 45; Pharm. Post. 20, 369; Chem. Drug. 1887, 388; Fortschritt. 1887, 301; Jsb. d. Pharm. 1887, 369; Chem. Centrbl. 1887, 312 and 971; Ber. d. chem. Ges. 20, 148 R.
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Pharm. Centralh. 28, 255; Chem. Centrbl. 1887, 732.
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U. S. P. 1890, p. 91.

FOR TOTAL ALKALOIDS.—To 20 gms. of cinchona in very fine powder and contained in a bottle with accurately ground glass stopper, add 200 c. c. of a previously prepared mixture of 19 volumes of alcohol, 5 vols. chloroform, and 1 vol ammonia water, stopper the bottle, and shake thoroughly and frequently during four hours. Then separate the liquid by pouring it into another bottle through a funnel containing a pellet of cotton so that no material loss by evaporation may result. Transfer 100 c. c. of the clear filtrate (representing 10 gms. cinchona) to a beaker, and

evaporate to dryness. Dissolve the residue of crude alkaloids thus obtained in 10 c. c. of water and 4 c. c. normal sulphuric acid, with the aid of gentle heat, filter the cooled solution into a separating funnel, and wash the beaker and filter until filtrate no longer has an acid reaction, using as small a quantity of water as possible. Now add 5 c. c. normal potassium hydrate to render decidedly alkaline, and extract alkaloids by shaking the mixture, first with 20 c. c., then repeatedly with 10 c. c. of chloroform, until a drop of the last chloroform extraction leaves no residue when evaporated on a watch glass. Evaporate the united chloroformic extracts in a tared beaker, dry the residue at 100° C. and weigh. The weight found, multiplied by 10 will give the percentage of total alkaloids in the specimen of cinchona tested.

FOR QUININE.—Transfer 50 c. c. of the clear filtrate remaining from the preceding process (and representing 5 gm. of cinchona) to a beaker, evaporate it to a dryness, and proceed as directed for total alkaloids, using only half the amounts of volumetric acid and alkali there directed. Add the united chloroformic extracts containing the alkaloids in solution, gradually, and in small portions at a time, to about 5 gm. of powdered glass contained in a porcelain capsule placed over a water bath, so that when the contents of the capsule are dry, all or nearly all of the dry alkaloids shall be in intimate admixture with the powdered glass, and the chloroform be completely expelled. Now moisten the residue with ether, and having placed a funnel containing a filter of a diameter of 7 c. m. and well wetted with ether over a small graduated tube (A), transfer the ether-moistened residue from the capsule. Rinse the latter several times, if necessary, with fresh ether, so as to transfer the whole of the residue to the filter, then percolate with ether added drop by drop, until exactly 10 c. c. of percolate have been obtained. Then collect another volume of 10 c. c. by similar slow percolation with ether, in a second graduated tube (B). Transfer the contents of the two tubes completely, (using ether for washing) to two small, tared capsules, properly marked (A and B) so as to avoid confusion, evaporate to a constant weight at 100° C. and weigh them. (The residue in A will contain practically all the quinine, together with a portion of the alkaloids less soluble in ether; the residue B will consist almost entirely of these alkaloids.) From the amount of residue obtained in capsule A deduct that contained in B, and multiply the remainder by 20. The product will represent, approximately, the percentage of quinine (containing 1 molecule of water) in the specimen of cinchona tested.

1890: (20) U. S. PHARMACOPŒIA. Assay of extract of *nuxvomica*.

U. S. P. 1890, p. 154.

Put 2 gm. of the dried extract of *nux vomica* into a glass separator, add to it 20 c. c. of a previously prepared mixture of 2 volumes of alcohol, 1 vol. ammonia water (s. g. 0.960), and 1 vol. water, and shake the separator till the extract is dissolved. Then add 20 c. c. of chloroform and agitate during five minutes. Allow the chloroform to separate, remove it as far as possible, pour into the separator a few c. c. of chloroform, and, without shaking, draw this off through the stop-cock to wash the outlet tube. Repeat the extraction with two further portions of chloroform of 15 c. c. each, and wash the outlet tube each time as just directed. Collect all the chloroformic solutions in a wide beaker, expose the latter to a gentle heat, on a water-bath, until the chloroform and ammonia are completely dissipated, add to the residue 10 c. c. of decinormal sulphuric acid measured from a burette, stir gently, and then add 20 c. c. of hot water. When solution has taken place add 2 c. c. of Brazil wood solution, and then carefully run in centinormal potassium hydrate, until a permanent pinkish color is produced. Divide the number of c. c. of centinormal potash used by 10, subtract the number found from the amount of decinormal acid used, multiply the remainder by 0.0364 and that product by 50 (or multiply at once by 1.82), which will give the percentage of total alkaloids in the extract, it being assumed that strychnine and brucine are present in equal proportion, and the above factor being found by taking the mean of their respective molecular weights $[(334 + 394) \div 2 = 364]$.

1890: (21) U. S. PHARMACOPŒIA. Assay of opium, crude, extract or tincture.

U. S. P. 1890, pp. 156, 292, and 424.

EXTRACT OF OPIUM.—Dissolve 4 grs. in 30 c. c. of water, filter the solution through a small filter, and wash the filter and residue with water, until all soluble matters are extracted, collecting the washings separately. Evaporate in a tared capsule, first the washings to a small volume, then add the first filtrate, and evaporate the whole to a weight of ten grams. Rotate the concentrated solution about in the capsule until the rings of extract are redissolved, pour the liquid into a tared Erlenmeyer flask having a capacity of 100 c. c., and rinse the capsule with a few drops of water at a time, until the entire solution weighs 15 gm. Then add 8.5 c. c. of alcohol, shake well, add 20 c. c. of ether and shake again. Now add 2.2 c. c. of ammonia water, stopper the flask with a sound cork, shake it thoroughly during ten minutes and set aside in a moderately cool place for six hours or over night.

CRUDE OPIUM.—Introduce the opium (which if fresh, should be in very small pieces, and if dry, in very fine powder) into a bottle having

a capacity of about 300 c. c. add 100 c. c. of water, cork it well, and agitate frequently during 12 hours. Then pour the whole as evenly as possible upon a wetted filter of 12 cm. diameter, and when the liquid has drained off, wash the residue with water, carefully dropped upon the edges of the filter and the contents, until 150 c. c. of filtrate are obtained. Then carefully transfer the moist opium back to the bottle by means of a spatula, add 50 c. c. of water, agitate thoroughly and repeatedly during 15 minutes and return the whole to the filter. When the liquid has drained off, wash the residue as before, until the second filtrate measures 150 c. c., and finally collect about 20 c. c. more of a third filtrate. Evaporate in a tared capsule, first, the second filtrate to a small volume, then add the first filtrate, rinsing the vessel with the third filtrate, and continue the evaporation until the residue weighs 14 gm. Rotate the concentrated solution about in the capsule until the rings of extract are redissolved, pour the liquid into a tared Erlenmeyer flask of 100 c. c. capacity, and rinse the capsule with a few drops of water at a time, until the entire solution weighs 20 gm. Then add 12.2 c. c. alcohol, shake well, add 25 c. c. of ether and shake again. Then add 3.5 c. c. ammonia water, stopper the flask, shake thoroughly during ten minutes and set aside in a moderately cool place for six hours or over night.

TINCTURE OF OPIUM.—Evaporate 100 c. c. to about 20 c. c., add 40 c. c. of water, mix thoroughly, and set aside for an hour, occasionally stirring, and disintegrating the resinous flakes adhering to the capsule. Then filter, and wash the filter and residue with water, until all soluble matters are extracted, collecting the washings separately. Evaporate in a tared capsule, first the washings to a small volume, then add the first filtrate, and evaporate the whole to a weight of 14 gm. Then proceed exactly as with crude opium from this point, using same quantities.

After standing for 6 hours or over night, remove the stopper carefully, and should any crystals adhere to it, brush them into the flask. Place in a small funnel two rapidly acting filters, of 7 cm. diameter, plainly folded, one within the other (the triple fold of the inner being laid against the single fold of the outer), wet them well with ether, and decant the ethereal solution as completely as possible upon the inner filter. Add 10 c. c. of ether to the contents of the flask, rotate it, and again decant the ethereal layer upon the inner filter. Repeat this operation with another portion of 10 c. c. of ether. Then pour into the filter the liquid in the flask, in portions, in such a way as to transfer the greater portion of the crystals to the filter, and, when this has passed through, transfer the remaining crystals to the filter by washing the flask with several portions of water, not using more than about 10 c. c.

in all. Allow the double filter to drain, then apply water to the crystals drop by drop, until they are practically free from mother-water, and afterwards wash them, drop by drop, from a pipette, with a saturated alcoholic solution of morphine. When this has passed through, displace the remaining alcohol by ether, using 10 c. c. or more if necessary. Allow the filter to dry in a moderately warm place, at a temperature not exceeding 60°, to constant weight, then carefully transfer the crystals to a weighed watch-glass, and weigh. The weight found represents percentage of morphine (crystallized) from tincture, multiplied by 10 gives percentage from crude opium, and multiplied by 25 percentage from extract.

1890. (22) NAGELVOORT, J. B. Rapid estimation of morphine in opium.

Am. J. Pharm. 1890, 407; *Terapia moderna*, 1891, 80; *Rep. de Pharm.* 1891, 283.

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J. Pharm. Chim. (5) 3, 593; *Jsb. d. Pharm.* 1891, 550; J. Chem. Soc. 60, 1043; *Chem. Centrbl.* 1891, ii, 233.

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Proc. A. Ph. A., 1891, 124;

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Proc. Mich. St. Ph. A. 1891, 67.

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Schweiz. Wochens. 29, 147; *Chem. Centrbl.* 1891, i, 1006; J. Chem. Soc. 60, 1402.

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J. Anal. Chem. 4, 390; *Chem. Centrbl.* 1891, i, 373; J. Chem. Soc. 60, 964; *Chem. News*, 63, 18.

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Pharm. Rund. 1891, 240; Apoth. Ztg. 1891, 537; Am. J. Pharm. 1892, 26.
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Ann. di Chim. e di Farm. **14**, 149; Einf. Stud. Alk. p. 542.
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J. Anal. Chem. **6**, 162; Chem. Centrbl. 1892, ii, 188.
- 1892: (2) ALLEN, A. H. Methyl orange as the best indicator for titration of alkaloids.
Chem. and Drug. 1892, 104; Proc. Am. Ph. A. 1892, 1035.
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Apot. Ztg. 1892, 435; Am. J. Pharm. 1892, 522; Chem. Centrbl. 1892, ii, 638.
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Compt. rend. 1892, **115**, 512; Chem. News, 1892, 223; Jsb. d. Pharm. 1892, 491; Proc. A. Ph. A. 1893, 817; Am. J. Pharm. 1892, 368; J. Chem. Soc. **64**, ii, 146.
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Compt. rend. **115**, 1085; J. Chem. Soc. **64**, ii, 199; Chem. Centrbl. 1893, i, 233.

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J. Chem. Soc. **64**, ii, 310.
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Ph. A. 1893, 404.
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Rep. de Pharm. 1894, 75.
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Chem. Centrbl. 1892, ii, 638.
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i, 235 and 322; J. Chem. Soc. **64**, ii, 397; Jsb. d. Pharm.
1893, 171; Am. J. Pharm. 1893, 78.
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721; Chem. Centrbl. 1893, i, 865; J. Chem. Soc. **64**, ii,
608.
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- 1895: (12) GERMAN PHARMACOPEIA: Assay of cinchona.
Arzneibuch f. d. deutsche Reich, 1895, p. 79.

20 gm. of finely powdered bark are shaken vigorously and repeatedly with 10 c. c. ammonia, 20 c. c. alcohol and 170 c. c. ether, and after 24 hours 100 c. c. of the clear liquid is decanted. Add to this 3 c. c. normal hydrochloric acid and 27 c. c. water, and distill off the ether and alcohol, and if necessary, add enough normal hydrochloric acid to make slightly acid. When cold, add 3 c. c. normal caustic potash, or enough to reddened phenolphthalein, and filter. The precipitate is washed repeatedly with small amounts of water until the washings are no longer alkaline (to phenolphthalein). Press gently with filter paper, and dry, first over sulphuric acid, then at 100° and weigh.

1895: (13) GERMAN PHARMACOPŒIA. Assay of opium.

Arzneibuch, f. d. deutsche Reich, 1895, p. 236.

Rub up in a mortar 6 gms. powdered opium with 6 c. c. water; dilute, and wash the mixture with water into a weighed flask, and make up to weight of 54 gms. Shake repeatedly during an hour, and filter through a plaited filter of 10 c. m. diameter. To 42 gm. of the filtrate add 2 gm. of a mixture of 17 parts ammonia and 83 parts water, mix well but avoid excessive shaking, and filter at once through plaited filter. 36 gms. of this filtrate is placed in a tared flask, 10 gm. ether is added, the flask is shaken, and 4 gm of the ammonia diluted as above, and the shaking continued until the liquid has cleared. It is then allowed to stand 6 hours. Pour off the ethereal layer as completely as possible on to a plain filter of 8 c. m. diameter, to the aqueous liquid add 10 gm. ether, agitate a few minutes and again pour off the ether. Then pour the water on the filter, at the same time transferring the crystals; wash twice with 5 gm. water saturated with ether. Dry the filter at 100°, transfer crystals completely to the flask. Dry to constant weight and weigh.

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Pharm. J. Trans. (4) 6, 368.

Shake 10 c. c. of the fluid extract with 10 c. c. of chloroform, 50 c. c. of water, and a decided excess of ammonia; separate the chloroformic solution and twice repeat the agitation and separation with chloroform. Shake the mixed chloroformic solutions with 5 c. c. of diluted sulphuric acid, mixed with twice its volume of warm water, separate, and repeat the operation. Wash the mixed acid liquids with 3 c. c. of chloroform, then agitate with 10 c. c. of chloroform and an excess of ammonia. Separate the chloroformic solution, twice repeat the agitation and separation with chloroform, wash the mixed chloroformic solutions with 5 c. c. of water containing 1 drop of solution of ammonia, evaporate on a water bath, dry the residue at 100° C., and weigh. Next dissolve the residue in 10 c. c. of decinormal hydrochloric acid, neutralize with centinormal sodium hydroxide, using tincture of cochineal as an indicator, deduct the measure of soda solution from 100 c. c., and multiply the remainder by .00287. The product will be the weight in grams of alkaloids present in 10 c. c. of fluid extract.

- 1898: (4) BRITISH PHARMACOPŒIA. Assay of cinchona, tincture or fluid extract.

Pharm. J. Trans. (4) 6, 368.

Place 5 c. c. of the fluid extract in a stoppered glass separator, together with 25 c. c. of water, 30 c. c. of benzolated amyl alcohol, and 15 c. c. of solution of potassium hydroxide. After shaking repeatedly, the lower dark-colored alkaline layer that separates is run off and again shaken with 30 c. c. of benzolated amyl alcohol, the lower layer that now separates being rejected. The two upper layers containing the alkaloids in solution are then mixed, washed with a little water, and agitated thoroughly with 30 c. c. of a warm mixture of 1 volume of diluted hydrochloric acid and 5 volumes of water. This operation is repeated with the alcoholic liquor that separates, and the two acid layers are subsequently mixed. The alkaloids are then shaken out with 10 c. c. of chloroform and sufficient solution of ammonia to impart a strongly alkaline reaction; the agitation and separation repeated with two successive

quantities of 10 c. c. of chloroform, the chloroformic liquids mixed, evaporated slowly, the residue dried at 110° C., and the residual alkaloids weighed.

1898: (5) BRITISH PHARMACOPŒIA. Assay of ipecac, fluid extract.

Pharm. J. Trans. (4) 6, 369.

Dilute 20 c. c. of the extract with an equal bulk of water, remove the alcohol by the aid of a water bath, add to the warm solution an excess of solution of lead subacetate, filter, wash the precipitate with water, and add the washings to the filtrate. Remove excess of lead from filtrate by precipitation with dilute sulphuric acid, filter, wash, and add the washings to the filtrate. Transfer filtrate to a separator, add excess of solution of ammonia (s. g. 0.959), and agitate with 25 c. c. of chloroform. Separate and set aside the chloroformic solution, and twice repeat the agitation with chloroform and the subsequent separation. Then mix the chloroformic solutions, evaporate, dry at a temperature below 80° C., and weigh the residue of total alkaloids.

1898: (6) BRITISH PHARMACOPŒIA. Assay of nux vomica, fluid extract.

Pharm. J. Trans. (4) 6, 369.

Evaporate 10 c. c. to a thick syrup on a water-bath, dissolve the residue in 20 c. c. of water, heating if necessary, place the solution in a separator, and shake out with 5 gm. of sodium carbonate dissolved in 25 c. c. of water, together with 10 c. c. of chloroform. Separate, and twice repeat the operation with chloroform only. Then mix 6 c. c. of dilute sulphuric acid with 25 c. c. of water, divide the mixture into three parts, and shake the mixed chloroformic solutions with each in turn. Mix the acid liquids, dilute with water to 175 c. c. and shake well and frequently in a stoppered flask during half an hour with 25 c. c. of potassium ferrocyanide solution (10 gms. in 200 c. c.) The precipitate formed is transferred to a filter, the flask rinsed with water containing one-fortieth its volume of dilute sulphuric acid and the precipitate washed till the washings are free from bitterness. Next shake the precipitate in a separator with 5 c. c. of solution of ammonia, add 15 c. c. of chloroform in two successive solutions, evaporate in a counterpoised dish in a current of warm air, the dish being covered to avoid loss of strychnine. The residue is dried for 1 hour on a water-bath, and then weighed.

1898: (7) BRITISH PHARMACOPŒIA. Assay of opium tincture.
Pharm. J. Trans. (4) 6, 380.

Evaporate 80 c. c. to 30 c. c. mixing the residue with 3 gm. of freshly-slaked lime, adding water to 85 c. c., and setting aside for half an hour, stirring occasionally. Next, filter off 50 c. c. of the tincture, add 5 c. c. of 90% alcohol and 30 c. c. of ether, shake, and add 2 gm. of ammonium chloride. Shake well and frequently during half an hour, set aside for 12 hours for the morphine to separate, filter, wash with morphinated water, and dry the crystals first by gentle pressure between filter paper, then at 55° or 60° C., and finally at 110° C. for two hours. Weigh the crystals, and titrate 0.3 gm. with decinormal solution of sulphuric acid until the liquid, after boiling, slightly reddens blue litmus paper. Add to the weight of anhydrous morphine, thus indicated, 0.05 gm. to cover loss.

PART II.—INDEX OF AUTHORS.

- ADRIAN and GALLOIS. 1887. (16) Opium.
ALESSANDRI. 1882. (5) Cinchona alkaloids.
ALLEN, A. H. 1891. (8) Aconite.
“ 1892. (2) Use of methyl orange.
“ 1892. (3) Volumetric estimation.
“ 1896. (8) Titration of quinine.
ARNDT, E. M. 1890. (18) Emetine.
BARTHE L. 1892. (5) Volumetric estimation.
“ 1892. (6) Quinine and cinchona alkaloids.
BARTHEL, G. and DIETERICH, E. 1888. (13) Morphine.
BECK, C. R. 1892. (17) Ipecac.
BECKURTS, H. 1887. (9) Morphine.
“ 1889. (2) Strychnine, brucine, and general process.
“ 1890. (5) Strychnine.
“ 1891. (9) Nux vomica, aconite, belladonna and hyoscyamus.
“ 1894. (4) General process.
“ 1896. (6) Hydrastine and berberine.
“ and FRERICHS. 1896. (2) General process.
“ “ HOLST. 1887. (25) Strychnine and brucine.
“ “ 1887. (26) Strychnine, alkalimetric.
“ “ SCHRAVT. 1887 (11) Morphine.
BERNHARDT, W. 1884. (2) Morphine.
BIEL, J. 1882. (6) Cinchona.

- BIEL, J. 1887. (21) Morphine.
 " 1888. (8) Nicotine.
- BIÉTRIX and CAZENEUVE. 1892. (15) Caffeine.
- BIRD, F. C. J. 1887. (10) Morphine.
 " 1892. (10) Emetine.
- BLUNT, T. P. 1889. (10) Emetine.
 " 1890. (13) Emetine, alkalimetric.
- BLYTH, A. W. 1877. (1) Caffeine.
 " 1881. (7) Quinine.
- BRAITHWAITE and FARR. 1886. (14) Morphine.
- BRUNNER, H. 1894. (8) Caffeine and theobromine.
 " and LEINS, H. 1893. (10) Caffeine and theobromine.
- CANNEPIN and VAN ELJK. 1893. (12) Morphine.
- CARLES. 1872. (2) Quinine.
- CASPARI, C. and DOHME, R. L. 1893. (17) Assay by titration.
- CAVENDONI. 1888. (14) General process.
- CAZENEUVE and CAILLOL. 1877. (2) Piperine.
 " " BIÉTRIX. 1892. (15) Caffeine.
- CHRISTIANSEN, A. 1890. (4) General process, iodometric.
- CLAASEN, E. 1890. (12) Codeine and morphine.
- COBLENTZ. 1885. (2) Atropine.
- CONROY, M. 1883. (4) Strychnine.
 " 1884. (1) Morphine.
- COWNLEY and PAUL. 1887. (27) Caffeine in coffee.
 " " " 1887. (28) Caffeine in tea.
- CRIPPS, R. A. 1887. (32) Coniine.
 " 1895. (5) Emetine.
 " and WITHEY, A. 1889. (17) Emetine.
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- DELACOUR. 1896. (19) Caffeine.
- DEVRIJ, J. E. 1880. (6) Quinine as herapathite.
 " 1882. (2) General process for barks.
 " 1882. (3) Quinine as herapathite.
 " 1885. (6) Quinine as oxalate.
 " 1885. (7) Cinchonidine in quinine sulphate.
 " 1885. (8) Quinine in sulphate.
 " 1885. (9) Cinchona alkaloids.
 " 1886. (12) Quinine as chromate.
- DE WIJS, B. C. J. 1893. (11) Morphine.
- DIETERICH, E. 1885. (18) Morphine.

- DIETERICH, E. 1887. (12) Morphine.
 " 1887. (24) Strychnine.
 " 1889. (1) Morphine.
 " 1890. (10) Morphine.
 " 1890. (11) Morphine and emetine.
 " 1894. (5) Discussion of methods.
 " and BARTHEL. 1888. (13) Morphine.
- DOHME, R. L. and CASPARI, C. 1893. (17) Assay by titration.
- DOMERGUE and NICOLAS. 1892. (14) Caffeine.
- DOTT, D. B. 1894. (12) Morphine, alkalimetric.
 " 1896. (21) Opium assay.
- DRAGENDORFF. 1874. (2) Strychnine.
 " 1878. (4) Theobromine.
- DUNSTAN, W. R. and SHORT. 1883. (5) Strychnine.
 " " 1883. (6) Strychnine and brucine.
 " and RANSOM. 1884. (7) Atropine.
 " " 1885. (1) Atropine.
 " and TICKLE. 1896. (27) Aconitine.
- DWARS. 1878. (5) Quinine
 ! 1879. (1) Quinine and strychnine.
- EGELING. 1890. (3) Strychnine.
- EMINGER, A. 1896. (20) Theobromine.
 " and HILGER. 1894. (9) Theobromine.
- ESCHENBURG, H. 1896. (24) Cinchona.
- EYKMAN. 1881. (8) Cinchona.
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- FARR, E. H. and BRAITHWAITE. 1886. (14) Morphine.
 " and WRIGHT, R. 1894. (2) Discussion of methods.
 " " 1897. (6) Estimation of alkaloids.
- FAWSSETT. 1889. (13) Cinchona.
- FLEURY, G. 1879. (8) Morphine.
- FLUECKIGER, F. A. 1879. (9) Morphine.
 " 1885. (17) Morphine.
 " 1886. (17) Emetine.
 " 1889. (5) Morphine.
- FORSTER and RIECHELMANN. 1897. (2) Caffeine.
- FOUQUET, L. 1897. (7) Codeine and morphine.
- FRERICHS, G. and BECKURTS. 1896. (2) General process.
- GALLOIS and ADRIAN. 1887. (16) Morphine.
- GANE. 1896. (16) Caffeine.

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GEROCK, J. E. 1889. (6) Brucine and strychnine.
GERRARD, A. W. 1882. (12) Atropine.
GÖEBEL, H. 1887. (17) Morphine.
" 1889. (3) Quinine and cinchonidine.
GÖECKEL and TRILLICH. 1898. (2) Caffeine.
GOMBERG, M. 1896. (17) Caffeine.
GRANDVAL, A., and LAJOUX, H. 1893. (6) Caffeine.
" " 1893. (18) General process.
" " 1893. (19) Emetine, cocaine, and cinchona.
" " 1897. (9) Morphine.
GROVES, T. B. 1864. General process.
GUILLOT. 1892. (16) Caffeine.
GUNN, A. 1896. (13) Coca alkaloids.
HAGER. 1880. (4) Cinchona alkaloids.
" 1881. (2) Alkaloids as picrates.
" 1883. (1) Method of extraction.
HAUBENSAK, W. 1891. (4) Cinchona.
HEGLAND, J. M. A. 1896. (7) Hydrastine.
HEINTZ, E. 1878. (2) Method of extraction.
HERETH, F. S. 1886. (1) Method of titration.
HESSE, O. 1880. (7) Quinine.
" 1881. (6) Cinchonidine.
HEUT, G. 1893. (13) Coniine and nicotine.
HILBIG, C. 1880. (5) Cinchona.
HILGER and EMINGER. 1894. (9) Theobromine.
" " JUCKENACK. 1897. (4) Caffeine.
HINSDALE, S. J. 1890. (4) Morphine.
HOLDERMAN. 1889. (9) Morphine, iodometric.
HOLST and BECKURTS. 1887. (25) Strychnine and brucine.
" " 1887. (26) Strychnine.
HOOPER, D. 1886. (10) Quinine.
HOWARD, D. 1896. (10) Quinine.
JANDONS, A. 1887. (20) Morphine.
JOHNSTONE, W. 1889. (11) Piperine.
JONES, H. W. 1886. (16) Emetine.
JUCKENACK and HILGER. 1897. (4) Caffeine.
KASPAR. 1886. (11) Cinchona.
KEBLER, L. F. 1895. (1) Morphine.

- KEBLER, L. F. 1896. (26) Cinchona.
 " and LA WALL. 1895. (7) Volumetric process.
- KELLER, C. C. 1892. (12) Emetine.
 " 1893. (1) Cinchona.
 " 1893. (2) Emetine.
 " 1893. (3) Emetine.
 " 1893. (4) Strychnine.
 " 1893. (7) Strychnine.
 " 1894. (1) General process.
 " 1894. (13) Hydrastine.
 " 1895. (9) Strychnine.
 " 1895. (11) Cocaine.
 " 1896. (3) General process.
 " 1897. (3) Caffeine.
- KERNER, G. 1880. (8) Quinine.
- KIPPENBERGER, K. 1896. (4) Separation of alkaloids from ptomaines.
 " 1896. (5) General iodometric process.
- KISSEL. 1882. (4) Cinchona.
- KISSLING, R. 1882. (10) Nicotine.
 " 1882. (11) Nicotine.
 " 1883. (2) Nicotine.
 " 1889. (9) Nicotine, polariscopic.
 " 1895. (3) Nicotine and ammonia.
- KOTTMAYER, G. 1892. (11) Emetine.
- KRAMERS, I. G. 1896. (9) Quinine.
- KREMEL, A. 1887. (3) Aconite.
 " 1887. (14) Morphine.
 " 1888. (1) Emetine, hydrastine and berberine.
 " 1888. (2) Strychnine.
 " 1888. (7) Caffeine.
 " 1891. (10) Coniine.
- KUNZE, W. E. 1894. (6) Caffeine and theobromine.
- KUERSTEINER, C. 1892. (8) Cinchona.
- LAJOUX, H. and GRANDVAL, A. 1893. (6) Caffeine.
 " " 1893. (18) General process.
 " " 1893. (19) Emetine, cocaine, and cinchona.
 " " 1897. (9) Morphine.
- LALIEN. 1885. (15) Morphine.
- LAMBERT, A. 1891. (1) Morphine.
- LANDRIN. 1889. (18) Cinchona.

- LANGLOIS and PORTES. 1881. (1) Morphine.
 LA WALL and KEBLER. 1895. (7) Comparison of indicators.
 LEINS, H. 1894. (14) Caffeine and theobromine.
 " and BRUNNER, H. 1893. (10) Caffeine and theobromine.
 LENZ, W. 1888. (4) Quinine, comparison of methods.
 LIEUNNIGH, F. 1893. (22) General process.
 LIGNON. 1887. (33) Emetine.
 LILLY. 1887. (2) Atropine, and general process.
 LLOYD, J. U. 1885. (11) Berberine.
 " 1891. (2) General process.
 LOOFF, G. 1890. (7) Morphine.
 " 1890. (8) Morphine.
 " 1896. (12) Morphine.
 LÖESCH, A. 1879. (5) General process.
 " 1879. (6) General process.
 " 1887. (31) Caffeine.
 LYNN, J. 1876. (3) Morphine.
 LYONS, A. B. 1884. (6) General process.
 " 1885. (12) Emetine.
 " 1885. (13) Strychnine.
 " 1886. (3) Berberine.
 " 1886. (4) Hydrastine.
 " 1886. (5) Physostigmine.
 " 1886. (6) Strychnine.
 " 1886. (7) Discussion of methods.
 " 1886. (8) Cocaine.
 " 1886. (9) Cinchona.
 MACEWEN. 1887. (34) Physostigmine.
 MARKOWNIKOFF, B. W. 1876. (2) Caffeine.
 MASING, E. 1877. (3) Veratrine and physostigmine.
 MAUPY, L. 1897. (5) Theobromine.
 MEINERT and WRAMPELMEIER. 1886. (18) Opium.
 MENDINI, A. 1895. (10) Emetine.
 MEYER, H. 1882. (7) Cinchona.
 MEYER, A. 1893. (5) Emetine.
 MONTEMARTINI and TRASCIATTI. 1897. (8) Morphine.
 MYLIUS, E. 1880. (9) Opium alkaloids.
 " 1881. (10) Morphine.
 NAGELVOORT, J. B. 1890. (22) Morphine.
 NEUMAN, S. 1889. (14) Quinine.

- NICHOLS, H. T., and NORTON, T. H. 1892. (1) Examination of Lloyd's method.
- NICOLAS and DOMERGUE. 1892. (14) Caffeine.
- PARTHEIL, A. 1892. (4) Iodo-eosin as indicator.
- PATRONOUILLARD. 1880. (3) Caffeine.
- PAUL, B. H. 1891. (6) Caffeine.
- “ and COWNLEY. 1887. (27) Caffeine in coffee.
- “ “ 1887. (28) Caffeine in tea.
- PETIT, A. 1879. (7) Morphine.
- “ and TERRAT, P. 1896. (18) Caffeine.
- PEZZOLATO, A. 1891 (7) Nicotine.
- PHARMACOPŒIA, British. 1885. (19) Cinchona.
- “ “ 1885. (20) Nux vomica.
- “ “ 1885. (21) Opium.
- “ “ 1898. (3) Belladonna.
- “ “ 1898. (4) Cinchona.
- “ “ 1898. (5) Ipecac.
- “ “ 1898. (6) Nux vomica.
- “ “ 1898. (7) Opium.
- “ U. S. 1890. (19) Cinchona.
- “ “ 1890. (20) Nux vomica.
- “ “ 1890. (21) Opium.
- “ German. 1895. (12) Cinchona.
- “ “ 1895. (13) Opium.
- PINETTE, J. 1892. (9) Nicotine.
- PLUGGE, P. C. 1887. (1) General volumetric process.
- “ 1887. (13) Separation of opium alkaloids.
- POEHL. 1881. (9) Pilocarpine.
- POPOVICI, M. 1889. (8) Nicotine.
- PORTES and LANGLOIS. 1881. (1) Morphine.
- PRESCOTT, A. B. 1877. (5) Quinine.
- “ 1879. (11) Morphine.
- “ 1880. (1) General process.
- “ 1896. (1) Periodides.
- “ and THUM. 1878. (1) Cinchona, comparison of methods.
- PROLIUS. 1881. (4) Cinchona.
- PRUNIER, L. 1879. (3) Cinchona.
- PUCKNER, W. A. 1896. (14) Caffeine.
- RANSOM, F. 1887. (5) Emetine.
- “ and DUNSTAN. 1884. (7) Belladonna.

- RANSOM, F. and DUNSTAN. 1885. (1) Belladonna.
 RANWEZ. 1893. (16) Aconite, hyoscyamus and cicuta.
 REDWORD. 1884. (8) Belladonna.
 RICHMOND and SEATON. 1890. (6) Quinine.
 RIECHELMANN and FORSTER. 1897. (2) Caffeine.
 ROTHER. 1880. (11) Opium.
 RUDDIMAN, E. A. 1887. (22) Quinine.

 SARNOW and SCHWEISSINGER. 1890. (2) General process.
 SCHACHT, C. 1881. (5) Quinine.
 SCHLÆFER, L. 1887. (23) Cinchonidine.
 SCHEFFER, E. 1885. (5) Nicotine.
 SCHEIBE, E. 1883. (7) Morphine.
 SCHEIBLER. 1886. (2) General process.
 SCHLICKUM, O. 1887. (15) Morphine.
 SCHMIDT, J. H. 1892. (7) Quinine.
 SCHMITT, E. 1880. (10) Opium.
 SCHRAGE, F. 1878. (3) Cinchona.
 SCHRAUT, A., and BECKURTS, H. 1887. (11) Morphine.
 SCHWEISSINGER. 1885. (14) Strychnine and brucine.
 " and SARNOW. 1890. (2) General process.
 SCHWICKERATH, K. 1894. (3) General process.
 SEATON and RICHMOND. 1890. (6) Quinine.
 SHIMOYANO, Y. 1883. (3) Cinchona.
 " 1885. (10) Cinchona.
 SHORT and DUNSTAN. 1883. (5) Strychnine.
 " " 1883. (6) Strychnine and brucine.
 SKALWEIT, J. 1881. (3) Nicotine.
 " 1882. (9) Nicotine.
 SMITH, E. D. 1887. (29) Caffeine.
 SMITH, C. E. 1896. (23) Nux vomica.
 SMITH, J. D., and TESCHEMACHER, E. F. 1888. (11) Morphine.
 SNOW, H. W. 1886. (13) Caffeine.
 " 1887. (6) Emetine.
 " 1888. (5) General process, Mayer's reagent.
 " 1888. (6) General process, phospho-molybdic acid.
 " 1889. (7) Strychnine and brucine.
 SOKOLOFF, N. 1876. (1) Caffeine.
 " 1893. (8) Caffeine.
 " 1895. (4) Caffeine.
 SPENCER, G. L. 1890. (15) Caffeine.
 " 1891. (5) Caffeine.

- SQUIBB, E. R. 1882. (1) Morphine.
 " 1882. (8) Cinchona.
 " 1884. (4) Cocaine.
 " 1884. (5) Caffeine.
 " 1885. (3) Atropine.
 " 1885. (4) Cocaine.
 " 1887. (4) Cocaine.
 " 1887. (19) Morphine.
 " 1888. (9) Cocaine.
 " 1888. (10) Morphine.
- STILWELL, C. M. 1887. (8) Morphine.
- STEDER, W. 1894. (10) Cortex granati.
- SUESS, P. 1890. (16) Theobromine.
 " 1893. (9) Theobromine.
- TASSILLY, E. 1897. (1) Caffeine.
- TERRAT, P., and PETIT, A. 1896. (18) Caffeine.
- TESCHEMACHER, E. F. 1877. (4) Morphine.
 " and SMITH, J. D. 1888. (11) Morphine.
- THIEL, J. 1894. (7) Theobromine.
- THOMPSON, F. A. 1890. (17) Conine.
 " 1891. (3) General process.
 " 1893. (15) Hydrastine and berberine.
- THRESH, J. C. 1880. (2) Volumetric method.
- THUM and PRESCOTT. 1878 (9) Cinchona.
- TICKLE and DUNSTAN. 1896. (27) Aconitine.
- TRASCIATTI and MONTEMARTINI. 1897. (8) Morphine.
- TRAUB. 1892. (13) General iodometric process.
- TRILLICH and GÖCKEL. 1898. (2) Caffeine.
- UMNEY, J. C. 1895. (6) Aconitine.
- VAN DER MARK. 1889. (12) Cocaine.
 " 1889. (16) Strychnine.
- VAN ELJK and CANNÉPIN. 1893. (12) Morphine.
- VAN ITALLIE. 1888. (15) Atropine.
 " 1889. (15) Alkalimetric process.
 " 1890. (1) Alkalimetric process.
 " 1895. (14) General process.
- VAN LEDDEN-HULSEBOSCH. 1893. (21) General process.
 " " 1895. (8) General process.
 " " 1896. (15) Caffeine.

- VAN ZWALUWENBERG, A. 1887. (7) Bibliography of opium assay.
VEDRÉDI, V. 1893. (14) Nicotine, comparison of methods.
" 1895. (2) Nicotine and ammonia.
" 1896. (11) Nicotine and ammonia.
VENTURINI, V. 1886. (15) Morphine, comparison of methods.
VITALI, D. 1893. (20) General process for chlorides and sulphates.
VITÉ, F. 1890. (14) Caffeine.
VON PERGER. 1884. (3) Morphine.

WAAGE, TH. 1887. (30) Caffeine.
WAGNER. 1861. Periodide method.
WAINWRIGHT. 1885. (16) Morphine.
WENTZKY, O. 1898. (1) Resumé of processes.
WIEDA. 1888. (3) Cinchona, resumé of 25 processes.
WILEY, C. D. 1887. (18) Morphine.
WILLIAMS, R. 1888. (12) Morphine.
WITHBY, A., and CRIPPS, R. A. 1889. (17) Emetine.
WOLFRAM, G. 1879. (4) Theobromine.
WRAMPMEIER and MEINERT. 1886. (18) Opium.
WRIGHT, R., and FARR, E. H. 1894. (2) Discussion of methods.
" " 1897. (6) General process.

YVON. 1879. (2) Morphine.

ZINOFFSKY. 1872. (1) General volumetric method.
" 1874. (1) Coniine.

PART III.—SUBJECT INDEX.

- ACONITINE. 1887. (3) Kremel.
" '91. (8) Allen.
" '95. (6) Umney.
" '96. (27) Dunstan and Tickle.
ATROPINE. 1882. (12) Gerrard.
" '84. (7) Dunstan and Ransom.
" '84. (8) Redword.
" '85. (1) Dunstan and Ransom.
" '85. (2) Coblenz.
" '85. (3) Squibb.
" '88. (15) Van Itallie.
" '98. (3) British Pharmacopolia.

- BERBERINE. 1885. (11) Lloyd.
 " '86. (3) Lyons.
 " '88. (1) Kremel.
 " '93. (15) Thompson.
 " '96. (6) Beckurts.
- BRUCINE. 1883. (6) Dunstan and Short.
 " '85. (14) Schweissinger.
 " '87. (25) Beckurts and Holst.
 " '89. (2) Beckurts.
 " '89. (6) Gerock.
 " '89. (7) Snow.
 " See also strychnine.
- CAFFEINE. 1876. (2) Markownikoff.
 " '77. (1) Blyth.
 " '80. (3) Patronouillard.
 " '84. (5) Squibb.
 " '86. (13) Snow.
 " '87. (27) Cownley and Paul.
 " '87. (28) Cownley and Paul.
 " '87. (29) E. D. Smith.
 " '87. (30) Waage.
 " '87. (31) Lœsch.
 " '88. (7) Kremel.
 " '90. (14) Vitè.
 " '90. (15) Spencer.
 " '91. (5) Spencer.
 " '91. (6) Paul.
 " '92. (14) Domergue and Nicolas.
 " '92. (15) Cazeneuve and Biètrix.
 " '92. (16) Guillot.
 " '93. (6) Grandval and Lajoux.
 " '93. (8) Sokoloff.
 " '93. (10) Brunner and Leins.
 " '94. (6) Kunze.
 " '94. (8) Brunner.
 " '94. (14) Leins, A.
 " '95. (4) Sokoloff.
 " '96. (14) Puckner.
 " '96. (15) Van Ledden-Hulsebosch.
 " '96. (16) Gane.
 " '96. (17) Gomberg.

CAFFEINE.	1896.	(18)	Petit and Terrat.
"	'96.	(19)	Delacour.
"	'96.	(25)	Georges.
"	'97.	(1)	Tassilly.
"	'97.	(2)	Forster and Riechelmann.
"	'97.	(3)	Keller.
"	'97.	(4)	Hilger and Juckenack.
"	'98.	(2)	Trillich and Gœckel.

CEPHAELIN, see IPECAC.

CINCHONA ALKALOIDS:

CINCHONIDINE.	1881.	(6)	Hesse.
"	'85.	(7)	De Vrij.
"	'87.	(23)	Schæfer.
"	'89.	(3)	Gœbel.
QUININE.	1872.	(2)	Carles.
"	'77.	(5)	Prescott.
"	'78.	(5)	Dwars.
"	'79.	(1)	Dwars.
"	'79.	(10)	——
"	'80.	(6)	De Vrij.
"	'80.	(7)	Hesse.
"	'80.	(8)	Kerner.
"	'81.	(7)	Blyth.
"	'82.	(3)	De Vrij.
"	'85.	(6)	De Vrij.
"	'85.	(8)	De Vrij.
"	'85.	(19)	British Pharmacopœia.
"	'86.	(10)	Hooper.
"	'86.	(12)	De Vrij.
"	'87.	(22)	Ruddiman.
"	'88.	(4)	Lenz.
"	'89.	(3)	Gœbel.
"	'89.	(14)	Neumann.
"	'90.	(6)	Seaton and Richmond.
"	'90.	(19)	U. S. Pharmacopœia.
"	'92.	(7)	J. H. Schmidt.
"	'95.	(12)	German Pharmacopœia.
"	'96.	(8)	Allen.
"	'96.	(9)	Kramers.
"	'96.	(10)	Howard.

TOTAL ALKALOIDS. 1878. (1) Prescott and Thum.

" '78. (3) Schrage.

CINCHONA, Continued:

TOTAL ALKALOIDS.	1879.	(3)	Prunier.
"	'80.	(4)	Hager.
"	'80.	(5)	Hilbig.
"	'81.	(4)	Prollius.
"	'81.	(5)	Schacht.
"	'81.	(8)	Eykman.
"	'82.	(2)	De Vrij.
"	'82.	(4)	Kissel.
"	'82.	(5)	Alessandri.
"	'82.	(6)	Biel.
"	'82.	(7)	H. Meyer.
"	'82.	(8)	Squibb.
"	'82.	(13)	Fairthorne.
"	'83.	(3)	Shimoyana.
"	'85.	(9)	De Vrij.
"	'85.	(10)	Shimoyana.
"	'86.	(9)	Lyons.
"	'86.	(11)	Kaspar.
"	'88.	(3)	Wieda.
"	'89.	(13)	Fawsett.
"	'89.	(18)	Landrin.
"	'91.	(4)	Haubensak.
"	'92.	(6)	Barthe.
"	'92.	(8)	Kuersteiner.
"	'93.	(1)	Keller.
"	'96.	(24)	Eschenburg.
"	'96.	(26)	Kebler.
"	'98.	(4)	British Pharmacopoeia.
COCAINE.	1884.	(4)	Squibb.
"	'85.	(4)	Squibb.
"	'86.	(8)	Lyons.
"	'87.	(4)	Squibb.
"	'88.	(9)	Squibb.
"	'89.	(12)	Van der Mark.
"	'95.	(11)	Keller.
"	'96.	(13)	Gunn.
CODEINE.	1887.	(13)	Plugge.
"	'90.	(12)	Claasen.
"	'97.	(7)	Fouquet.
CONIINE.	1874.	(1)	Zinoffsky.
"	'76.	(1)	Sokoloff.
"	'87.	(32)	Cripps.

- CONINE 1890. (17) Thompson.
 " '91. (10) Kremel.
 " '93. (13) Heut.

EMETINE, see IPECAC.

ESERINE, see PHYSOSTIGMINE.

- GENERAL PROCESSES. 1861. Wagner.
 " '64. Groves.
 " '78. (2) Heintz.
 " '79. (5) Lœsch.
 " '79. (6) Lœsch.
 " '80. (1) Prescott.
 " '81. (2) Hager.
 " '83. (1) Hager.
 " '84. (6) Lyons.
 " '86. (2) Scheibler.
 " '87. (2) Lilly.
 " '88. (5) Snow.
 " '88. (14) Cavendoni.
 " '89. (2) Beckurts.
 " '91. (2) Lloyd.
 " '91. (3) Thompson.
 " '91. (9) Beckurts.
 " '92. (1) Nichols and Norton.
 " '93. (16) Ranwez.
 " '93. (18) Grandval and Lajoux.
 " '93. (19) Grandval and Lajoux.
 " '93. (21) Van Ledden-Hulsebosch.
 " '93. (22) Lieunnigh.
 " '94. (1) Keller.
 " '94. (2) Farr and Wright.
 " '94. (3) Schwickerath.
 " '94. (4) Beckurts.
 " '94. (5) Dieterich.
 " '95. (8) Van Ledden-Hulsebosch.
 " '95. (14) Van Itallie.
 " '96. (1) Prescott.
 " '96. (2) Beckurts and Frerichs.
 " '96. (3) Keller.
 " '96. (4) Kippenberger.
 " '97. (6) Farr and Wright.
 " '98. (1) Wentzky.

GENERAL PROCESSES, VOLUMETRIC.	'72.	(1)	Zinoffsky.
"	"	'80.	(2) Thresh.
"	"	'86.	(1) Hereth.
"	"	'86.	(7) Lyons.
"	"	'87.	(1) Plugge.
"	"	'88.	(6) Snow.
"	"	'89.	(15) Van Itallie.
"	"	'90.	(1) Van Itallie.
"	"	'90.	(2) Schweissinger and Sarnow.
"	"	'90.	(4) Christiansen.
"	"	'92.	(2) Allen.
"	"	'92.	(3) Allen.
"	"	'92.	(4) Partheil.
"	"	'92.	(5) Barthe.
"	"	'92.	(13) Traub.
"	"	'93.	(7) Keller.
"	"	'93.	(17) Caspari and Dohme.
"	"	'93.	(20) Vitali.
"	"	'95.	(7) Kebler and LaWall.
"	"	'96.	(5) Kippenberger.

HYDRASTINE.	1886.	(4)	Lyons.
"	'88.	(1)	Kremel.
"	'93.	(15)	Thompson.
"	'94.	(13)	Keller.
"	'96.	(6)	Beckurts.
"	'96.	(7)	Hegland.

HYOSCYAMINE, see ATROPINE.

IPECAC.	1885.	(12)	Lyons.
"	'86.	(16)	H. W. Jones.
"	'86.	(17)	Flückiger.
"	'87.	(5)	Ransom.
"	'87.	(6)	Snow.
"	'87.	(33)	Lignon.
"	'88.	(1)	Kremel.
"	'89.	(10)	Blunt.
"	'89.	(17)	Cripps and Withby.
"	'90.	(11)	Dieterich.
"	'90.	(13)	Blunt.
"	'90.	(18)	Arndt.
"	'92.	(10)	Bird.

IPECAC.	1892.	(11)	Kottmayer.
"	'92.	(12)	Keller.
"	'92.	(17)	Beck.
"	'93.	(2)	Keller.
"	'93.	(3)	Keller.
"	'93.	(5)	A. Meyer.
"	'95.	(5)	Cripps.
"	'95.	(10)	Mendini.
"	'96.	(22)	—
"	'98.	(5)	British Pharmacopæia.

MORPHINE	1877.	(4)	Teschemacher.
"	'76.	(3)	Lynn.
"	'79.	(2)	Yvon.
"	'79.	(7)	Petit.
"	'79.	(8)	Fleury.
"	'79.	(9)	Flückiger.
"	'79.	(11)	Prescott.
"	'80.	(9)	Mylius.
"	'80.	(10)	E. Schmitt.
"	'80.	(11)	Rother.
"	'81.	(1)	Portes and Langlois.
"	'81.	(10)	Mylius.
"	'82.	(1)	Squibb.
"	'83.	(7)	Scheibe.
"	'84.	(1)	Conroy.
"	'84.	(2)	Bernhardt.
"	'84.	(3)	Von Perger.
"	'85.	(15)	Lalien.
"	'85.	(16)	Wainwright.
"	'85.	(17)	Flückiger.
"	'85.	(18)	Dieterich.
"	'85.	(21)	British Pharmacopœia.
"	'86.	(14)	Braithwaite and Farr.
"	'86.	(15)	Venturini.
"	'86.	(18)	Wrampelmeier and Meinert.
"	'87.	(7)	Van Zwaluwenberg.
"	'87.	(8)	Stillwell.
"	'87.	(9)	Beckurts.
"	'87.	(10)	Bird.
"	'87.	(11)	Beckurts and Schraut.
"	'87.	(12)	Dietrich.

- MORPHINE. 1887. (13) Plugge.
 " '87. (14) Kremel.
 " '87. (15) Schlickum.
 " '87. (16) Adrian and Gallois.
 " '87. (17) Gœbel.
 " '87. (18) C. D. Wiley.
 " '87. (19) Squibb.
 " '87. (20) Jandons.
 " '87. (21) Biel.
 " '88. (10) Squibb.
 " '88. (11) Teschemacher and Smith.
 " '88. (12) Williams.
 " '88. (13) Dietrich and Barthel.
 " '89. (1) Dietrich.
 " '89. (4) Holderman.
 " '89. (5) Flückiger.
 " '90. (7) Looff.
 " '90. (8) Looff.
 " '90. (9) Hinsdale.
 " '90. (10) Dietrich.
 " '90. (11) Dietrich.
 " '90. (12) Claasen.
 " '90. (21) U. S. Pharmacopœia.
 " '90. (22) Nagelvoort.
 " '91. (1) Lambert.
 " '93. (11) de Wijs.
 " '93. (12) Cannepin and Van Eijk.
 " '94. (12) Dott.
 " '95. (1) Kebler.
 " '95. (13) German Pharmacopœia
 " '96. (12) Looff.
 " '96. (21) Dott.
 " '97. (7) Fouquet.
 " '97. (8) Montemartini and Trasciatti.
 " '97. (9) Grandval and Lajoux.
 " '98. (7) British Pharmacopœia.

NARCOTINE. 1897. (13) Plugge.

- NICOTINE. 1881. (3) Skalweit.
 " '82. (9) Skalweit.
 " '82. (10) Kissling.
 " '82. (11) Kissling.

NICOTINE.	1883.	(2)	Kissling.
"	'85.	(5)	Scheffer.
"	'88.	(8)	Biel.
"	'89.	(8)	Popovici.
"	'89.	(9)	Kissling.
"	'91.	(7)	Pezzolato.
"	'92.	(9)	Pinette.
"	'93.	(13)	Heut.
"	'93.	(14)	Vedrödi.
"	'95.	(2)	Vedrödi.
"	'95.	(3)	Kissling.
"	'96.	(11)	Vedrödi.

OPIUM ALKALOIDS, separation. 1887. (13) Plugge.

PHYSOSTIGMINE.	1877.	(3)	Masing.
"	'86.	(5)	Lyons.
"	'87.	(34)	MacEwan.

PILOCARPINE. 1881. (9) Pöehl.

PIPERINE. 1877. (2) Cazeneuve and Caillol.

" '89. (11) Johnstone.

POMEGRANATE (cortex granati). 1894. (10) Stöder.

QUININE, see CINCHONA.

STRYCHNINE.	1874.	(2)	Dragendorff.
"	'79.	(1)	Dwars.
"	'83.	(4)	Conroy.
"	'83.	(5)	Dunstan and Short.
"	'83.	(6)	Dunstan and Short.
"	'85.	(13)	Lyons.
"	'85.	(14)	Schweissinger.
"	'85.	(20)	British Pharmacopœia.
"	'86.	(6)	Lyons.
"	'87.	(24)	Dieterich.
"	'87.	(25)	Beckurts and Holst.
"	'87.	(26)	Beckurts and Holst.
"	'88.	(2)	Kremel.
"	'89.	(2)	Beckurts.
"	'89.	(6)	Geroock.
"	'89.	(7)	Snow.
"	'89.	(16)	Van der Mark.

STRYCHNINE.	1890.	(3)	Egeling.
"	'90.	(4)	Keller.
"	'90.	(5)	Beckurts.
"	'90.	(20)	U. S. Pharmacopœia.
"	'93.	(4)	Keller.
"	'94.	(11)	Cushman.
"	'95.	(9)	Keller.
"	'96.	(23)	C. E. Smith.
"	'98.	(6)	British Pharmacopœia.

THEBAINE, see OPIUM.

THEINE, see CAFFEINE.

THEOBROMINE.	1878.	(4)	Dragendorff.
"	'79.	(4)	Wolfram.
"	'90.	(16)	Süss.
"	'93.	(9)	Süss.
"	'93.	(10)	Brunner and Leins.
"	'94.	(6)	Kunze.
"	'94.	(7)	Thiel.
"	'94.	(8)	Brunner.
"	'94.	(9)	Hilger and Eminger.
"	'94.	(14)	Leins.
"	'96.	(20)	Eminger.
"	'97.	(5)	Maupy.

VERATRINE. 1877. (3) Masing.

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